

No. 94293-5

SUPREME COURT OF THE STATE OF WASHINGTON

PUGET SOUNDKEEPER ALLIANCE,

Petitioner,

v.

STATE OF WASHINGTON, DEPARTMENT OF ECOLOGY; and
STATE OF WASHINGTON, POLLUTION CONTROL HEARINGS
BOARD,

Respondents.

**BRIEF OF AMICI CURIAE NORTHWEST PULP & PAPER
ASSOCIATION, ASSOCIATION OF WASHINGTON BUSINESS
AND ASSOCIATION OF WASHINGTON CITIES**

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TABLE OF CONTENTS

I. INTRODUCTION.....	1
II. IDENTITY AND INTEREST OF AMICI CURIAE.....	3
III. STATEMENT OF THE CASE	3
IV. ARGUMENT	3
A. Neither the Court nor the Board Has Authority to Grant the Relief Sought by Soundkeeper	3
B. EPA Approval of Test Methods through Formal Rulemaking Ensures That Affected Parties Have the Opportunity for Notice and Comment	6
C. The Clean Water Act Requires That Where States Impose More Stringent Standards Than EPA They Must Do So Based on Sound Science and with EPA Approval	8
D. Overturning the Court of Appeals Decision Could Potentially Bring Washington’s NPDES Permitting Program to a Standstill	11
V. CONCLUSION	13

Appendix A

Clean Water Act Methods Update Rule for the Analysis of Effluent, 82 Fed. Reg. 40,836 (Aug. 28, 2017) (to be codified at 40 C.F.R. pt. 136) (excerpts).

TABLE OF AUTHORITIES

Cases

<i>Akiak Native Community v. EPA</i> , 625 F.3d 1162 (9 th Cir. 2010)	10
<i>Arkansas v. Oklahoma</i> , 503 U.S. 91, 112 S. Ct. 1046, 117 L. Ed. 2d 239 (1992).....	9
<i>City of Abilene v. EPA</i> , 325 F.3d 657 (5 th Cir. 2003)	9
<i>International Paper Co. v. Ouellette</i> , 479 U.S. 481, 107 S. Ct. 805, 93 L. Ed. 2d 883 (1987).....	10
<i>Nat'l Ass'n of Home Builders v. Defenders of Wildlife</i> , 551 U.S. 644, 127 S. Ct. 2518, 168 L. Ed. 2d 467 (2007).....	10
<i>New York v. United States</i> , 505 U.S. 144, 112 S. Ct. 2408, 120 L. Ed. 2d 120 (1992).....	8, 9
<i>PUD No. 1 of Jefferson County v. Washington Dept. of Ecology</i> , 511 U.S. 700, 114 S. Ct. 1900, 128 L. Ed. 2d 716 (1994).....	9
<i>Puget Soundkeeper Alliance v. Dep't of Ecology</i> , 197 Wn. App. 1078, 2017 WL 702504 (2017)	1, 2, 5
<i>Puget Soundkeeper Alliance v. Dept. of Ecology</i> , PCHB No. 13-137c, Findings of Fact, Conclusions of Law and Order (July 23, 2015)	1

Statutes

33 U.S.C. § 1313(b), (c)	9
33 U.S.C. § 1314(a)(1)	9
33 U.S.C. § 1342(b).....	9, 10
33 U.S.C. § 1342(c)(3)	10
33 U.S.C. § 1342(d).....	10
33 U.S.C. § 1342(d)(4)	10
RCW 90.48.520	2

Regulations

40 C.F.R. § 123.44.....	10
40 C.F.R. § 123.63.....	10
40 C.F.R. § 123.64.....	10
40 C.F.R. § 131.11(a)(1).....	9
40 C.F.R. § 131.11(b).....	9
40 C.F.R. part 136.....	2, 4, 5, 12
40 C.F.R. § 136.1.....	4
40 C.F.R. § 136.4.....	5, 12
40 C.F.R. § 136.4(a).....	12
40 C.F.R. § 136.4(a)(4).....	12
40 C.F.R. § 136.5.....	5, 12, 13
40 C.F.R. § 136.5(c).....	12
40 C.F.R. § 136.5(c)(3).....	12
40 C.F.R. § 136.5(c)(5).....	12
40 C.F.R. § 136.6.....	5, 12
40 C.F.R. § 136, App. A.....	4
WAC 173-201A-260(3)(h).....	passim
75 Fed. Reg. 58,024 (Sept. 23, 2010).....	7
77 Fed. Reg. 29,758 (May 18, 2012).....	4, 7
77 Fed. Reg. 29,763 (May 18, 2012).....	7
80 Fed. Reg. 8,956 (Feb. 19, 2015).....	4
82 Fed. Reg. 40,836 (Aug. 28, 2017).....	4, 6, 7

82 Fed. Reg. 40,876 (Aug. 28, 2017)	6
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I. INTRODUCTION

In this brief, amici curiae Northwest Pulp & Paper Association (“NWPPA”), Association of Washington Business (“AWB”), and Association of Washington Cities (“AWC”) (collectively “Associations”) address the issue of whether the Washington Department of Ecology correctly required the use of Method 608 in the NPDES permit issued to Seattle Iron and Metals (“SIM”) to measure compliance with limits on the discharge of polychlorinated biphenyls (“PCBs”). On appeal brought by Puget Soundkeeper Alliance (“Soundkeeper”), the Pollution Control Hearings Board concluded that Ecology’s use of Method 608 was proper because WAC 173-201A-260(3)(h) requires that Ecology use an EPA-approved test method, and Method 608 is the only test method approved by EPA. *Puget Soundkeeper Alliance v. Dept. of Ecology*, PCHB No. 13-137c, Findings of Fact, Conclusions of Law and Order (July 23, 2015) (“Board Decision”) at 34-35.¹

The Board’s ruling was subsequently affirmed by the Division II Court of Appeals in an unpublished opinion. *Puget Soundkeeper Alliance v. Dep’t of Ecology*, 197 Wn. App. 1078, 2017 WL 702504 (2017) (“Opinion”). The Court of Appeals focused on the language of the applicable federal and

¹ A copy of the Board Decision is attached as Appendix B to the Department of Ecology’s Answer to Petition for Review.

state regulations in concurring with the Board and Ecology that Method 608 is the only test method approved by EPA, and thus the only test method available to Ecology for use in the SIM Permit.²

This Court subsequently granted Soundkeeper's petition for discretionary review. In its petition Soundkeeper virtually abandoned all pretense of basing its argument on an interpretation of WAC 173-201A-260(3)(h) and 40 C.F.R. part 136, the state and federal regulations mandating the Clean Water Act test methods to be used to measure compliance with NPDES permits. Instead, Soundkeeper appeals to broad policies behind RCW 90.48.520 and asserts that the Court of Appeals "with virtually no analysis or reasoning" held a "preference" for Ecology's "narrow interpretation" of WAC 173-201A-260(3)(h). Puget Soundkeeper Alliance's Petition for Discretionary Review ("Petition") at 17-18. *See* Puget Soundkeeper Alliance's Supplemental Brief ("Soundkeeper Supplemental Brief") at 7, 11. However, it is unclear what "interpretation" of WAC 173-

² The Court of Appeals rejected Soundkeeper's argument that Ecology could have used Method 1668C for PCB testing because Method 1668C is a "superseding method. . . published" under WAC 173-201A-260(3)(h). 2017 WL 702504 at * 6-7. Although Soundkeeper attempts to resurrect this argument in its Supplemental Brief, as noted in Ecology's Answer to Petition for Review, Soundkeeper did not raise the issue in its Petition for Review and it is therefore not properly before this Court. Ecology Answer to Petition for Review at 9 n.4. Should this Court decide to consider the argument, the Associations support Ecology's discussion of the issue in Ecology's Supplemental Brief.

201A-260(3)(h) Soundkeeper would have Ecology use. Soundkeeper's very request for relief, which asks this Court to order Ecology to deny SIM's NPDES permit unless Ecology obtains approval from EPA for the use of Method 1668C, acknowledges that Ecology is required to use a test method approved by EPA. The Court of Appeals properly rejected Soundkeeper's arguments and upheld the Board's decision.

II. IDENTITY AND INTEREST OF AMICI CURIAE

Northwest Pulp & Paper Association, the Association of Washington Business, and the Association of Washington Cities are described in the Associations' Motion for Leave to File Amici Curiae Brief. The Associations and their members have an interest in ensuring that the methods for testing PCBs used in NPDES permits under which they operate are based on sound science and have been reviewed and approved by EPA in accordance with state and federal law.

III. STATEMENT OF THE CASE

The Court of Appeals decision sets forth the relevant facts.

IV. ARGUMENT

A. Neither the Court Nor the Board Has Authority to Grant the Relief Sought by Soundkeeper.

Having conceded that EPA must approve a test method before it can be written into an NPDES permit by Ecology, and that EPA has not approved Method 1668C, Soundkeeper is left to argue that Ecology should be forced to

seek and obtain EPA approval of Method 1668C. Soundkeeper makes this argument despite its own acknowledgment that Ecology's authority to seek approval for an alternate test method is wholly discretionary, and that the Board lacks authority to require Ecology to seek such approval.

EPA's regulations set out approved test methods and require that such methods "shall . . . be used" by states in administering and issuing NPDES permits. 40 C.F.R. § 136.1.³ Method 608 is the only method approved by EPA for testing PCBs. 40 C.F.R. § 136, App. A. In addition to directing that Ecology must use test methods either approved by EPA or superseding versions of such methods, WAC 173-201A-260(3)(h) provides that Ecology "*may* also approve other [test] methods following consultation with adjacent states *and with the approval of the USEPA.*" (emphasis added).⁴ EPA's

³ Soundkeeper attached an outdated 2003 version of 40 C.F.R. part 136 to its Petition. The regulations were revised in 2012, and Soundkeeper has attached the 2012 version to its Supplemental Brief. 77 Fed. Reg. 29,758 (May 18, 2012). On August 28, 2017, EPA issued a Final Rule further updating the regulations. 82 Fed. Reg. 40,836 (Aug. 28, 2017) (excerpts attached as Appx. A to this amici brief). EPA had proposed the changes for public comment on February 19, 2015. 80 Fed. Reg. 8,956 (Feb. 19, 2015).

⁴ The amicus curiae brief filed by the Squaxin Island Tribe in support of Soundkeeper's Petition misrepresents WAC 173-201A-260(3)(h) as "allowing for the use of other laboratory methods as long as Ecology consults with EPA (not even requiring approval of EPA)." Amicus Curiae Squaxin Island Tribe Brief in Support of Puget Soundkeeper Alliance's Petition for Discretionary Review at 18. This is incorrect. The plain language of the regulation requires "the approval of the USEPA." The referenced "consultation" is with other states. Soundkeeper does not dispute that under

regulations set out the detailed procedures by which a state or other entity may apply to EPA for approval of an alternate test method. 40 C.F.R. §§ 136.4, 136.5, 136.6.

Soundkeeper admitted before the Court of Appeals that it is wholly up to Ecology to decide whether to seek approval from EPA for an alternate test method, stating that “requesting EPA’s permission” to use an alternate test method “is Ecology’s choice to make.” Petitioner’s Court of Appeals Reply Brief at 16. Nor has Soundkeeper challenged the Board’s finding, upheld by the Court of Appeals, that the Board lacks the authority to require Ecology to petition EPA for such approval. Board Decision at 35 (¶ 7), 48 (¶ 29); 2017 WL 702504 at * 7 n. 13. Accordingly, Soundkeeper does not ask this Court to compel Ecology to seek EPA’s approval for use of Method 1668c. Nor does Soundkeeper ask this Court to compel Ecology to issue SIM an NPDES permit that uses Method 1668C. Instead, before the Court of Appeals, Soundkeeper sought to prohibit Ecology from issuing SIM’s permit unless Ecology both applies for and obtains approval from EPA for the use of Method 1668C. Petitioner’s Court of Appeals Opening Brief at 46, 49-50; Petitioner’s Court of Appeals Reply Brief at 17. Similarly, Soundkeeper now asks this Court to instruct Ecology to “either deny permit issuance or

both WAC 173-201A-260(3)(h) and 40 C.F.R. part 136, the test method used by Ecology must be approved by EPA.

condition permit issuance on EPA's approval. . . to use of Method 1668C. . ."

Soundkeeper Supplemental Brief at 20.

However expressed, Soundkeeper's convoluted request for relief is tantamount to asking the Board to do the very thing that Soundkeeper admits the Board does not have the authority to do. Directing that the Board order Ecology to deny SIM's permit unless Ecology obtains EPA approval for Method 1668C is a back door attempt to mandate that Ecology exercise its discretionary authority to seek approval of the method. Moreover, there is no reason to suppose that EPA would in fact approve Method 1668C should Ecology ask it to do so. In fact, the evidence is quite to the contrary. As described below, EPA has already taken up and declined to approve Method 1668C in its 2010 proposed rule and 2012 final rule. Less than a week ago, EPA reiterated in its new Final Rule that Method 1668C has not been approved for use by EPA. 82 Fed. Reg. 40,836, 40,876 (Aug. 28, 2017). Soundkeeper's request for relief is nothing more than an attempt to force Ecology to seek and somehow obtain approval from EPA for a test method that EPA has already considered and failed to approve once. Neither the Board – nor this Court – have the authority to grant such relief.

B. EPA Approval of Test Methods through Formal Rulemaking Ensures That Affected Parties Have the Opportunity for Notice and Comment.

Under the Clean Water Act, approval of test methods by EPA are a matter of formal rulemaking, affording interested parties notice and the

opportunity to comment. *See, e.g.*, Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Analysis and Sampling Procedures, 75 Fed. Reg. 58,024 (Sept. 23, 2010). EPA's test method regulations have been revised many times through such rulemaking. As an example, the recent 2017 revisions approve new versions of previously approved EPA test methods, and in doing so EPA considered numerous comments and made changes based on such comments. 82 Fed. Reg. 40,836 (Aug. 28, 2017).

The importance of notice and comment rulemaking in the test methods development process is particularly obvious here, where EPA proposed in 2010 to approve Method 1668C and then declined to do so in its 2012 final rule. 75 Fed. Reg. 58,024 (Sept. 23, 2010); 77 Fed. Reg. 29,758 (May 18, 2012). EPA noted in the final rule that it had received comments from thirty-five separate individuals or organizations regarding the proposed approval of Method 1668C, thirty of which were critical of the method. 77 Fed. Reg. 29,763 (May 18, 2012).

According to EPA, "commenters opposing the method provided a detailed critique of the method, the inter-laboratory study, the peer reviews and the other supporting documentation." *Id.* Although stating that its decision did not negate the merits of the method, EPA noted several different criticisms of Method 1668C raised by the comments, including the use of poor data not fit for use in a comprehensive interlaboratory study, deviation

from existing guidelines, failure to consider the problem of background contamination, and failure to include all matrices in the test method validation study. *Id.* The statutory and regulatory framework requiring EPA test method approval through formal rulemaking allows for EPA consideration of such comments prior to approving monitoring test methods, ensuring that the required test methods are appropriate, reliable, and scientifically defensible.⁵

C. The Clean Water Act Requires That Where States Impose More Stringent Standards Than EPA They Must Do So Based on Sound Science and with EPA Approval.

Soundkeeper correctly notes that the Clean Water Act allows states to establish more stringent standards than those set by EPA. Petition at 13-14. But states are not required to do so, and their discretion is not unfettered. When states choose to exercise their discretion to establish more stringent standards, the CWA requires that – just as with the test method requirement at issue here – states must comply with statutes and regulations ensuring that their decision-making is subject to EPA review and approval and is based on sound science.

The U.S. Supreme Court has described the CWA as “a program of cooperative federalism.” *New York v. United States*, 505 U.S. 144, 167, 112

⁵ Ecology’s Supplemental Brief at § III.A, and its Court of Appeals Response Brief at 23-24, summarize the evidence before the Board of Ecology’s own concerns regarding the use of Method 1668C.

S. Ct. 2408, 120 L. Ed. 2d 120 (1992). The CWA “anticipates a partnership between the States and the Federal Government, animated by a shared objective. . . .” *City of Abilene v. EPA*, 325 F.3d 657, 659 (5th Cir. 2003) (quoting *Arkansas v. Oklahoma*, 503 U.S. 91, 101, 112 S. Ct. 1046, 117 L. Ed. 2d 239 (1992)). The Act thus sets out distinct roles for the federal and state governments. *PUD No. 1 of Jefferson County v. Washington Dept. of Ecology*, 511 U.S. 700, 704, 114 S. Ct. 1900, 128 L. Ed. 2d 716 (1994). For example, although states may adopt water quality standards more stringent than those required by EPA, states must submit proposed standards to EPA, and the CWA reserves for EPA the authority to approve or disapprove state-adopted water quality standards, to regularly review and approve or disapprove any revisions to those state standards, and under certain circumstances, to promulgate EPA’s own water quality standards. 33 U.S.C. § 1313(b), (c).

States developing water quality criteria must do so pursuant to EPA guidelines “accurately reflecting the latest scientific knowledge.” 33 U.S.C. § 1314(a)(1). Such criteria “must be based on sound scientific rationale.” 40 C.F.R. § 131.11(a)(1). And in establishing criteria states must establish numerical values based on EPA guidance or “other scientifically defensible methods.” 40 C.F.R. § 131.11(b).

Furthermore, EPA delegates administration of NPDES permits to a state only after review and approval of the state’s program. 33 U.S.C.

§ 1342(b). Although EPA cannot dictate the terms of state-administered NPDES permits, it retains oversight over state NPDES programs. A state must advise EPA of each permit it proposes to issue, and EPA may object to any individual permit that does not comply with the requirements of the CWA. *Nat'l Ass'n of Home Builders v. Defenders of Wildlife*, 551 U.S. 644, 650 n. 1, 127 S. Ct. 2518, 168 L. Ed. 2d 467 (2007); *International Paper Co. v. Ouellette*, 479 U.S. 481, 489, 107 S. Ct. 805, 93 L. Ed. 2d 883 (1987); *Akiak Native Community v. EPA*, 625 F.3d 1162, 1165 (9th Cir. 2010); 33 U.S.C. § 1342(b), (d); 40 C.F.R. § 123.44. If the state cannot address EPA's concerns, authority over the permit reverts to EPA. *Nat'l Ass'n of Home Builders* at 650 n. 1; 33 U.S.C. § 1342(d)(4). In addition, if a state is not administering its NPDES program in accordance with the CWA, EPA may withdraw its approval of the program as a whole. *Akiak Native Community*, 625 F.3d at 1165; 33 U.S.C. § 1342(c)(3); 40 C.F.R. §§ 123.63, 123.64.

The CWA test method regulations, along with Ecology's own regulation, ensure that the test methods written into Washington's NPDES permits are scientifically defensible and approved by EPA. Contrary to Soundkeeper's assertions, Ecology cannot simply ignore these regulations and use a test method that has not been and might never be approved by EPA. Ecology isn't "deferring" to EPA's compliance monitoring method. See Petition at 17. Ecology is following CWA regulations requiring that EPA

review and approve of a test method before that method is required in an NPDES permit.

D. Overturning the Court of Appeals Decision Could Potentially Bring Washington's NPDES Permitting Program to a Standstill.

Ecology manages several hundred NPDES individual permits such as the SIM permit at issue here; as well as general permits issued by Ecology for large groups of dischargers, including the Industrial Stormwater General Permit, Construction Stormwater General Permit, Phase I and II Municipal Permits, and the Boatyard General Permit. The Industrial Stormwater General Permit alone covers more than 1,000 facilities.⁶ None of these permits requires compliance with water quality standards using Method 1668C. If the Court were to overturn the Court of Appeals, and the Board was ordered to prohibit Ecology from denying the SIM permit unless Ecology obtains approval of Method 1668C from EPA, there would be nothing to stop Soundkeeper from subsequently asserting that Ecology must deny every permit requiring measurement of PCBs unless the permit requires monitoring with Method 1668C. In fact, Soundkeeper might very well argue that any permits currently requiring Method 608 are invalid. Ruling in

⁶ Industrial Stormwater General Permit Fact Sheet (May 7, 2014) at <http://www.ecy.wa.gov/programs/wq/stormwater/industrial/ISGPDraft2015FactSheet.pdf>

Soundkeeper's favor thus might have the bizarre result of rendering invalid existing permits using Method 608 – even though it is undisputed that Method 608 is approved by EPA in 40 C.F.R. part 136 and used by Ecology pursuant to WAC 173-201A-260(3)(h).

Moreover, the relief sought by Soundkeeper could bring Ecology's NPDES permit program to a virtual standstill while Ecology is required to use time and resources to seek approval of Method 1668C from EPA using the procedures set out in 40 C.F.R. § § 136.4 – 136.6. Among other things, applicants must submit a detailed description of the proposed alternate test procedure, together with references to published or other studies confirming the applicability of the alternate test procedure for the analysis of the effluents in question. 40 C.F.R. § § 136.4(a); 136.5(c). Applications for the use of alternate test procedures for regional use must provide a justification for using the alternate test procedure rather than procedures already approved by EPA. 40 C.F.R. § 136.5(c)(3). Applicants must also provide comparability data for the performance of the proposed alternate test procedure compared to the performance of the reference method. 40 C.F.R. § 136.4(a)(4); 136.5(c)(5). Approval of an alternate test procedure further requires compliance with the method modifications and analytical requirements set out in 40 C.F.R. § 136.6. And of course, as explained above, given EPA's consideration and failure to approve Method 1668C in

the past, there is no guarantee that after going through this process Ecology would in fact receive EPA approval of Method 1668C from EPA.

In the meantime, Ecology would be unable to move forward with other NPDES permitting, delaying water quality improvement efforts that would normally occur as part of the adaptive process ensuring that each permit cycle maintains and improves water quality in Washington. And the regulated community – both private and public entities – would be unable to plan and manage their operations with any sense of regularity and predictability. Such a result would be contrary not only to the CWA and its regulations but to the policies it seeks to further.⁷

V. CONCLUSION

For the foregoing reasons, amici Northwest Pulp and Paper Association, Association of Washington Business, and Association of Washington Cities respectfully request that this Court affirm the Court of Appeals decision in this case.

⁷ EPA's regulations allow "any person" to request EPA approval of an alternate test procedure. 40 C.F.R. § 136.5. Rather than attempting to force Ecology to exercise its discretionary authority to seek approval of Method 1668C, Soundkeeper could make its own application to EPA for approval of Method 1668C.

Respectfully submitted this 1st day of September, 2017.

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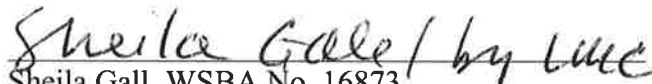
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APPENDIX A

Excerpts from

Clean Water Act Methods Update Rule for the Analysis of Effluent,

82 Fed. Reg. 40,836 (Aug. 28, 2017)

ENVIRONMENTAL PROTECTION AGENCY

40 CFR Part 136

[EPA-HQ-OW-2014-0797; FRL-9957-24-OW]

RIN 2040-AF48

Clean Water Act Methods Update Rule for the Analysis of Effluent

AGENCY: Environmental Protection Agency (EPA).

ACTION: Final rule.

SUMMARY: This rule modifies the testing procedures approved for analysis and sampling under the Clean Water Act. The changes adopted in this final rule fall into the following categories: New and revised EPA methods (including new and/or revised methods published by voluntary consensus standard bodies (VCSB), such as ASTM International and the Standard Methods Committee); updated versions of currently approved methods; methods reviewed under the alternate test procedures (ATP) program; clarifications to the procedures for EPA approval of nationwide and limited use ATPs; and amendments to the procedure for determination of the method detection limit to address laboratory contamination and to better account for intra-laboratory variability. **DATES:** This regulation is effective on September 27, 2017. The incorporation by reference of certain publications listed in the rule is approved by the Director of the Federal Register as of

September 27, 2017. For judicial review purposes, this final rule is promulgated as of 1:00 p.m. (Eastern time) on September 12, 2017 as provided at 40 CFR 23.2 and 23.7.

ADDRESSES: EPA has established a docket for this action under Docket ID No. EPA-HQ-OW-2014-0797. All documents in the docket are listed on the www.regulations.gov Web site. Although listed in the index, some information is not publicly available, e.g., confidential business information (CBI) or other information whose disclosure is restricted by statute. Certain other materials, such as copyrighted material are not placed on the Internet and will be publicly available only in hard copy form. Publicly available docket materials are available either electronically through www.regulations.gov or in hard copy at the Water Docket in EPA Docket Center, EPA/DC, EPA West William J. Clinton Building, Room 3334, 1301 Constitution Ave. NW., Washington, DC. The Public Reading Room is open from 8:30 a.m. to 4:30 p.m., Monday through Friday, excluding legal holidays. The telephone number for the Public Reading Room is 202-566-1744 and the telephone number for the Water Docket is 202-566-2426.

FOR FURTHER INFORMATION CONTACT: Adrian Hanley, Engineering and Analysis Division (4303T), Office of Water, Environmental Protection Agency, 1200 Pennsylvania Ave. NW., Washington, DC 20460-0001; telephone:

202-564-1564; email: hanley.adrian@epa.gov.

SUPPLEMENTARY INFORMATION:

A. General Information

1. Does this Action apply to me?

EPA proposed the changes in this method update rule for public comment on February 19, 2015 (80 FR 8956).

EPA Regions, as well as States, Territories and Tribes authorized to implement the National Pollutant Discharge Elimination System (NPDES) program, issue permits with conditions designed to ensure compliance with the technology-based and water quality-based requirements of the Clean Water Act (CWA). These permits may include restrictions on the quantity of pollutants that may be discharged as well as pollutant measurement and reporting requirements. If EPA has approved a test procedure for analysis of a specific pollutant, the NPDES permittee must use an approved test procedure (or an approved alternate test procedure if specified by the permitting authority) for the specific pollutant when measuring the required waste constituent. Similarly, if EPA has established sampling requirements, measurements taken under an NPDES permit must comply with these requirements. Therefore, entities with NPDES permits will potentially be affected by the actions in this rulemaking.

Entities potentially affected by the requirements of this rule include:

Category	Examples of potentially affected entities
State, Territorial, and Indian Tribal Governments	States, territories, and tribes authorized to administer the National Pollutant Discharge Elimination System (NPDES) permitting program; states, territories, and tribes providing certification under CWA section 401; state, territorial, and tribal owned facilities that must conduct monitoring to comply with NPDES permits.
Industry	Facilities that must conduct monitoring to comply with NPDES permits.
Municipalities	Publicly Owned Treatment Works (POTWs) or other municipality owned facilities that must conduct monitoring to comply with NPDES permits.

This table is not exhaustive, but rather provides a guide for readers regarding entities likely to be affected by this action. This table lists types of entities that EPA is now aware of that could potentially be affected by this action. Other types of entities not listed in the table could also be affected. To determine whether your facility is affected by this action, you should carefully examine the applicability language at 40 CFR 122.1 (NPDES purpose and scope), 40 CFR 136.1 (NPDES permits and CWA) and 40 CFR 403.1 (pretreatment standards purpose and applicability). If you have questions regarding the applicability of this action

to a particular entity, consult the appropriate person listed in the preceding **FOR FURTHER INFORMATION CONTACT** section.

B. What process governs judicial review of this rule?

Under Section 509(b)(1) of the Clean Water Act (CWA), judicial review of this CWA rule may be obtained by filing a petition for review in a United States Circuit Court of Appeals within 120 days from the date of promulgation of this rule. For judicial review purposes, this final rule is promulgated as of 1 p.m. (Eastern time) on September 12, 2017 as provided at 40 CFR 23.2.

Section 509(b)(2) provides that any rule (or requirements of any rule) for which review could have been obtained under Section 509(b)(1) may also not be challenged later in civil or criminal proceedings for enforcement.

C. Abbreviations and Acronyms Used in the Preamble and Final Rule Text

4AAP: 4-Aminoantipyrine
AA: Atomic Absorption
ADMI: American Dye Manufacturers Institute
AOAC: AOAC International
ASTM: ASTM International
ATP: Alternate Test Procedure
BOD₅: 5-day Biochemical Oxygen Demand test
CAS: Chemical Abstract Services

corrected an analyte name to 2,2'-oxybis(1-chloropropane), which matches the CAS Number 108-60-1.

EPA Method 624.1, Purgeables by GC/MS. This method measures purgeable organic pollutants in industrial discharges and other environmental samples by gas chromatography (GC) combined with mass spectrometry (MS), as provided under 40 CFR 136.1.

EPA Method 625.1, Base/Neutrals and Acids by GC/MS. This method measures semivolatile organic pollutants in industrial discharges and other environmental samples by GC/MS, as provided under 40 CFR 136.1.

2. EPA Methods 1600, 1603, 1680, and 1682

This rule implements the following changes for EPA microbiological methods 1600, 1603, 1680, and 1682 that correct typographical or other errors that EPA identified in the methods after publication. This rule revises all of these methods with new EPA document numbers and dates.

EPA Method 1600 for Enterococci using membrane filtration: In Table 3 Verification controls, EPA changed the negative control for brain heart infusion broth incubated at 45 °C from *Escherichia coli* to *Enterobacter aerogenes*. *E. coli* is thermotolerant and *E. aerogenes* is not, so *E. coli* is not an appropriate negative control when heated.

EPA Method 1603 for *E. coli* using membrane filtration: In section 11.5, EPA changed the number of colonies on a countable plate from 20–60 to 20–80 colonies. Sixty colonies was a typographical error. In addition, the following sentence was inadvertently omitted and EPA included it: Sample volumes of 1–100 mL are normally tested at half-log intervals (e.g., 100, 30, 10, and 3 mL).

EPA Method 1680 for fecal coliforms using multiple tube fermentation: In section 3.1 Definitions, the sentence “The predominant fecal coliform is *E. coli*.” now reads “The predominant fecal coliform can be *E. coli*.”

EPA Method 1682 for *Salmonella* by MSRV medium: (1) In section 9.3, Table 2, the lab-prepared spike acceptance criteria now reads: “Detect–254%” and “Detect–287%” and (2) in section 14.5, Table 9, the spiked *Salmonella* for Example 2, Liquid now reads “ 3.7×10^6 CFU/mL.”

B. Methods Incorporated by Reference

Currently, hundreds of methods and ATPs are incorporated by reference within 40 CFR part 136. In most cases, 40 CFR part 136 contains multiple approved methods for a single pollutant

and regulated entities often have a choice in the selected method. This rule incorporates by reference revisions to methods from two VCSBs: Standard Methods and ASTM. The VCSB methods in this rule are in compliance, as discussed more fully in Section IV.I below, with the National Technology Transfer Act which directs EPA to use voluntary consensus standards so long as they are consistent with applicable law and not otherwise impractical. The methods are available on their respective VCSB Web sites to everyone at a cost determined by the VCSB, generally from \$40 to \$80. Both organizations also offer memberships or subscriptions that allow unlimited access to their methods. The cost of obtaining these methods is not a significant financial burden for a discharger or environmental laboratory, making the methods reasonably available. This rule also includes USGS methods and vendor ATPs that are incorporated by reference. The ATPs and USGS methods are available free of charge on the Web site for that organization. Therefore, EPA concludes that the methods and Alternate Test Procedures (ATPs) incorporated by reference are reasonably available. The individual standards are discussed in greater detail below.

C. New Standard Methods and New Versions of Approved Standard Methods in 40 CFR 136.3

This rule approves new versions of currently approved Standard Methods. The new versions of currently approved Standard Methods clarify or improve the instructions in the method, improve the QC requirements, or make editorial corrections. Consistent with the previous method update rule (77 FR 29758, May 18, 2012), EPA generally approves and includes in 40 CFR part 136 only the most recent version of a method published by the Standard Methods Committee by listing only one version of the method with the year of publication designated by the last four digits in the method number (e.g., SM 3111 B–2011). The date indicates the latest revision date of the method. This allows use of a specific method in any edition that includes a method with the same method number and year of publication.

Most of the revisions included to Standard Methods in this rule do not contain any substantive changes. Each Standard Method entry contains the Standard Methods number and date, the parameter, and a brief description of the analytical technique. The methods listed below are organized according to

the table at 40 CFR part 136 in which they appear.

The following identifies new versions of previously approved Standard Methods that EPA is including in Table IB at 40 CFR part 136. Where there are substantive changes to the method, these are noted:

1. SM 2120 B–2011, color, platinum cobalt visual comparison method.
2. SM 2120 F–2011, color, ADMI weighted-ordinate spectrophotometer method. EPA previously approved this method as SM 2120 E–1993. It is also similar to the currently approved National Council for Air and Stream Improvement, Inc. method that uses American Dye Manufacturers Institute weighted-ordinate spectrophotometric parameters. A footnote on the method specifies that the pH should be 7.6 and not 7.0 when used for NPDES monitoring purposes, since the original method was approved with a reference pH of 7.6. Additionally, the currently approved methods for the Color parameter are assigned more specific parameter names.
3. SM 2130 B–2011, turbidity, nephelometric method.
4. SM 2310 B–2011, acidity, titration using electrometric endpoint or phenolphthalein endpoint.
5. SM 2320 B–2011, alkalinity, electrometric or colorimetric titration to pH 4.5.
6. SM 2340 B–2011 and SM 2340 C–2011, hardness, by the calculation method or EDTA titration.
7. SM 2510 B–2011, conductivity, Wheatstone bridge method.
8. SM 2540 B–2011, SM 2540 C–2011, SM 2540 D–2011, SM 2540 E–2011, and SM 2540 F–2011, total, filterable, non-filterable, volatile, and settleable residue (solids, listed in the same order as the method numbers), all by gravimetric methodologies.
9. SM 2550 B–2010, temperature, thermometric.
10. SM 3111 B–2011, SM 3111 C–2011, SM 3111 D–2011, and SM 3111 E–2011, metals, direct aspiration atomic absorption (AA) methods with different gas mixtures. Each method has a different list of metals; these lists were not changed.
11. SM 3112 B–2011, metals, applicable to mercury, cold-vapor atomic absorption spectrometric method.
12. SM 3113 B–2010, metals, electrothermic atomic absorption spectrometric method. The only substantive change is a reduction in the required replicate analyses of each calibration standard from three to two. Similar EPA methods do not require replicates of each calibration standard.

SM 9222 B–2006. This method analyzes Coliform (total) in the presence of chlorine. The newer method includes a number of technology updates that do not significantly change the procedure. In addition, the method:

a. Modified the procedure to allow for the use of a humidified incubator if loose-lidded plates are used during incubation.

b. Added a note that five typical and five atypical colonies per membrane need to be identified during coliform verification.

c. Moved the definition of “Coliform” that was Section 4 of SM 9222, and renumbered the rest of the document, such that the “Procedure” is now Section 4, instead of Section 5. This is not a substantive change except that in Table IA, Parameter 4 “Coliform (total), in presence of chlorine, number per 100 mL” the citation for “MF with enrichment” will be changed from “9222 (B+B.5c)–1997” to “9222 (B+B.4c)–2006.”

2. This rule replaces the membrane filtration method SM 9222 D–1997 with SM 9222 D–2006. This method analyzes Coliform (fecal) and Coliform (fecal) in the presence of chlorine. The new method allows use of a dry recirculating incubator as specified in the culture dishes section. In addition, this rule adds the following footnote to Tables IA and IH regarding SM 9222 D–2006 for fecal coliform verification frequency: “The verification frequency is at least five typical and five atypical colonies per sampling site on the day of sample collection & analysis.” SM 9222 D–2006 specifies that the fecal coliform colonies should be verified “at a frequency established by the laboratory,” which can be as low as zero. Colonies need to be verified to prevent misidentification of results as false positive or false negative.

3. This rule replaces the membrane filtration method SM 9222 G–1997 with SM 9222 G–2006 in Table IH. These methods analyze for *E. coli* and Fecal Coliforms. The newer method includes a number of technology updates that do not significantly change the procedure. In addition, the method now has a modified composition of EC broth to include different quantities of KH_2PO_4 and 4-methylumbelliferyl- β -D-glucuronide.

D. New Versions of Approved ASTM Methods in 40 CFR 136.3

This rule approves new versions of currently approved ASTM methods, for the same reasons outlined in the first paragraph of Section II.B above. Many of the new versions of ASTM Methods approved in 40 CFR part 136 do not

contain any substantive changes. Each entry contains (in the following order): Approved ASTM method number and date, the parameter, a brief description of the analytical technique. Where there were substantive changes, they are identified. The methods listed below are organized according to the table at 40 CFR part 136 in which they appear.

The following identifies new versions of currently approved ASTM methods that are included in Table IB at 40 CFR part 136:

1. ASTM D 511–09 (A, B), calcium and magnesium, titrimetric ethylenediamine tetraacetic acid (EDTA), AA direct aspiration.
2. ASTM D 516–11, sulfate ion, turbidimetric.
3. ASTM D 858–12 (A–C), manganese, atomic absorption (AA) direct aspiration, AA furnace.
4. ASTM D 859–10, silica, colorimetric, manual.
5. ASTM D 1067–11, acidity or alkalinity, electrometric endpoint or phenolphthalein endpoint; electrometric or colorimetric titration to pH 4.5, manual.
6. ASTM D 1068–10 (A–C), iron, AA direct aspiration; AA furnace; colorimetric (phenanthroline).
7. ASTM D 1126–12, hardness, titrimetric (EDTA).
8. ASTM D 1179–10 (A, B), fluoride ion, electrode, manual; colorimetric, (SPADNS).
9. ASTM D 1246–10, bromide ion, electrode.
10. ASTM D 1687–12 (A–C), chromium (total) and dissolved hexavalent chromium, colorimetric (diphenyl-carbazide); AA direct aspiration; AA furnace.
11. ASTM D 1688–12 (A–C), copper, AA direct aspiration, AA furnace.
12. ASTM D 1691–12 (A, B), zinc, AA direct aspiration.
13. ASTM D 1976–12, dissolved, total-recoverable, or total elements, inductively coupled plasma/atomic emission spectroscopy (ICP/AES).
14. ASTM D 3223–12, total mercury, cold vapor, manual.
15. ASTM D 3373–12, vanadium, AA furnace.
16. ASTM D 3557–12 (A–D), cadmium, AA direct aspiration, AA furnace, voltammetry.
17. ASTM D 3590–11 (A, B), total Kjeldahl nitrogen, manual digestion and distillation or gas diffusion; semi-automated block digester colorimetric (distillation not required).
18. ASTM D 4382–12, barium, AA furnace.
19. ASTM D 4658–09, sulfide ion, ion selective electrode.

20. ASTM D 5257–11, dissolved hexavalent chromium, ion chromatography.

21. ASTM D 5673–10, dissolved elements and total-recoverable elements, ICP/MS.

22. ASTM D 5907–13, filterable matter (total dissolved solids) and nonfilterable matter (total suspended solids), gravimetric, 180 °C gravimetric, 103–105 °C post washing of residue.

23. ASTM D 6508–10, inorganic anions (fluoride, bromide, chloride, nitrite, nitrate, orthophosphate, and sulfate), capillary ion electrophoresis with indirect UV detection.

24. ASTM D 7284–13, total cyanide, manual distillation with MgCl_2 followed by flow injection, gas diffusion amperometry.

25. ASTM D 7511–12, total cyanide, segmented flow injection, in-line ultraviolet digestion, followed by gas diffusion amperometry.

EPA has changed Table IC at 40 CFR part 136 as follows:

1. ASTM D 7065–11, nonylphenol, bisphenol A, *p*-tert-octylphenol, nonylphenol monoethoxylate, nonylphenol diethoxylate, gas chromatography/mass spectrometry (GC/MS).

E. New United States Geological Survey (USGS) Methods in 40 CFR 136.3

1. This rule adds USGS Methods I–2547–11 and I–2548–11 titled “Colorimetric Determination of Nitrate Plus Nitrite in Water by Enzymatic Reduction, Automated Discrete Analyzer Methods,” to Table IB for the analytes nitrate, nitrite, and combined nitrate-nitrite. Method I–2548–11 is a low level (analytical range) version of Method I–2547–11. Both methods are included in the same method title. The method can be found in USGS Survey Techniques and Methods, Book 5, Chapter B8. The method is available at no cost from the USGS Web site. This method follows the same procedure as in ATP Case No. N07–0003—Nitrate Elimination Company Inc.’s (NECi) Method N07–0003, Revision 9.0, March 2014, “Method for Nitrate Reductase Nitrate-Nitrogen Analysis,” which EPA approved in this rule.

F. New ATPs in 40 CFR 136.3

This rule approves six methods submitted to EPA for review through the alternate test procedures (ATP) program and deemed acceptable based on the evaluation of documented method performance.

The following ATP has nationwide approval for wastewater and is incorporated into Table IA:

tube" with Standard Method 9230B–2007.

4. This rule revises a hardness entry in Table IB to state "Ca plus Mg as their carbonates, by any approved method for Ca and Mg (See Parameters 13 and 33), provided that the sum of the lowest point of quantitation for Ca and Mg is below the NPDES permit requirement for Hardness." Previously, this was only allowed for inductively coupled plasma or AA direct aspiration Ca and Mg methods. The rationale behind this change is that if one calcium and magnesium method approved by EPA can be used to calculate hardness, then other EPA approved methods should also be permitted to do so.

5. This rule deletes "p 14" from footnote 24 of Table IB because the method is not on that page.

6. This rule deletes Method 200.5, in Table IB from the cobalt, molybdenum and thallium entries. These analytes have not undergone formal testing by this method, and this method should not have been approved for these analytes.

7. This rule removes the reference to costs in 40 CFR 136.3(b) because costs are not included in the referenced documents.

8. This rule removes the first instance of "are" in 40 CFR 136.3(e) because it is a typographical error.

I. Changes to Table II at 40 CFR 136.3(e) to Required Containers, Preservation Techniques, and Holding Times

This rule revises Table II at 40 CFR 136.3(e) as follows.

1. The rule adds rows to Table II that specify holding times for total/fecal coliforms, and fecal streptococci in Table IH. Previously the holding times for these bacterial tests were unspecified. Now these methods have the same holding time requirements as the other bacterial tests.

2. This rule changes the sodium thiosulfate concentrations in Table II for bacterial tests from 0.0008% sodium thiosulfate to 0.008%. EPA proposed this change in its last update to 40 CFR part 136 (75 FR 58066–58067), but inadvertently omitted it in the publication of the final rule.

3. The rule re-inserts language that was accidentally deleted from footnote 5 of Table II during the previous update to 40 CFR part 136. Footnote 5 now reads "ASTM D7365–09a specifies treatment options for samples containing oxidants (e.g., chlorine) for cyanide analysis. Also, Section 9060A of Standard Methods for the Examination of Water and Wastewater (20th and 21st editions) addresses dechlorination procedures for

microbiological analyses." Previously, the words: "for microbiological analyses," were not present, so the footnote did not specify that treatment options for samples containing oxidants is specifically for cyanide analysis, and that the dechlorination procedures are specifically for microbiological analyses.

4. EPA requested public comment on how to approve variances to sample preservation, containers or holding times listed in Table II for specific dischargers. Currently, 40 CFR 136.3(e) grants authority to either the permitting authority in the Region or the Regional ATP Program Coordinator to grant exceptions to Table II for a specific discharger.

Of the eight comments received, four commenters thought that the permitting authority should have the sole authority to approve these variance requests. Three commenters thought that the Regional ATP Program Coordinators should have sole authority to approve variance requests, and one commenter thought that the best approach was for the permitting authority and the Regional ATP Program Coordinator to approve Table II variances for specific dischargers collaboratively. Each of these commenters provided sound reasoning for their suggested approach to the review and approval of these types of requests.

EPA has chosen to defer any decision on revising the current language and to leave 40 CFR 136.3(e) unchanged in this final rule.

J. Clarifications/Corrections to ATP Procedures in 40 CFR 136.4, 136.5 and Allowed Modifications in 136.6

40 CFR 136.4 and 136.5 describe EPA procedures for obtaining approval to use an alternate test procedure either on a national basis, or for limited use by dischargers or facilities specified in the approval. In the 2012 Method Update Rule, EPA made several clarifying changes to the language of these sections. At the same time, however, in many places in 40 CFR 136.4 and 136.5 where the phrase "Regional Alternate Test Procedures Coordinator" or "Regional ATP Coordinator" appears, EPA inadvertently also inserted the phrase "or permitting authority" following the phrase. This error resulted from the use of the "search and replace" function on the computer. The effect of the change was to inadvertently authorize State permitting authorities to approve ATPs for limited use within the State. EPA never intended this result, as is demonstrated by two facts. First, in its proposal for the 2012 Update (75 FR 58024, September 23, 2010), EPA did

not propose to authorize State NPDES permitting authorities to approve limited use ATPs. Second, the rule states that the approval may be restricted to specific dischargers or facilities, or to all dischargers or facilities "specified in the approval for the Region." (emphasis added). This language evidenced EPA's intent that only the Region—not the State—would be authorized to issue any such limited use ATP approval. Finally, as further evidence of EPA's intent, in several places, the text of the rule only makes sense if read to authorize only the Regional ATP Coordinator, not the State permitting authority, to approve limited use ATPs. For example, 40 CFR 136.5(d)(1) provides that after a review of the application by the Alternate Test Procedure Regional ATP Coordinator or permitting authority, the Regional ATP Coordinator or permitting authority notifies the applicant and the appropriate State agency of approval or rejection of the use of the alternate test procedure. As previously written, if the State is acting on a request for approval, the regulation would require the State to inform itself of its own action in approving or rejecting the ATP, a superfluous requirement.

This rule deletes all instances of "or permitting authority" from 40 CFR 136.4 and 136.5 to correct this error and revise the rule text to its original intent. Based on this revision, EPA and EPA alone has the authority to approve limited use ATPs.

This rule also changes 40 CFR 136.4 and 136.5 to clarify the process for nationwide ATP approvals and the Regional ATP Coordinator's role in limited use ATP approvals. These changes do not significantly change the process; the intent is to make the text simpler and clearer.

Finally, this rule adds language to 40 CFR 136.6(b)(1) to clarify that if a method user is uncertain whether or not a modification is allowed under 40 CFR 136.6, the user should contact either their Director or EPA Regional ATP Coordinator.

K. Changes to Appendix B to 40 CFR Part 136—Definition and Procedure for the Determination of the Method Detection Limit (MDL)

EPA is revising the procedure for determination of the MDL primarily to address laboratory blank contamination and to better account for intra-laboratory variability. The MDL procedure has not been revised since it was originally promulgated in 1983. The suggestion for these revisions came first from The National Environmental Laboratory Accreditation Conference (NELAC)

D. Change to Method Modifications and Analytical Requirements in § 136.6, Methods Modification Paragraph

For clarification purposes, the following two lines have been added to the methods modification paragraph (b): Where the laboratory is using a vendor-supplied method, it is the QC criteria in the reference method, not the vendor's method that must be met to show equivalency. Where a sample preparation step is required (*i.e.*, digestion, distillation), QC tests are to be run using standards treated in the same way as samples.

Also in this paragraph, the paragraph (b)(4)(xvi), "Changes are allowed in purge-and-trap sample volumes or operating conditions," was incorrectly deleted and is being reinstated.

Further, paragraph (b)(4)(xvii), regarding allowable modifications to Method 625, is being deleted as Method 625 has been replaced in its entirety with an updated version with this rulemaking.

E. Changes to EPA Method 608.3

EPA received numerous comments on Method 608.3, ranging from pointing out minor typographical errors to questioning substantive technical aspects of the proposed method. In response, EPA revised the method to address many of those comments. See the Response to Comments document available in the electronic docket listed in the ADDRESSES section at the beginning of this document for a detailed description of the changes.

Additionally, based on comments received in response to the proposal, EPA is reverting to the MDL values in the earlier version of Method 608 for those analytes that were included in Table 1 of Method 608.3. The MDLs in the proposed version of 608.3 were chosen for the proposed revision because they were determined with a capillary GC column. However, as noted by commenters, the values are not derived from a multiple laboratory validation study. Therefore, EPA has restored the original Method 608 MDL values. At such time as EPA develops new multi-laboratory MDL and ML values for the method, they will be included in a future revision and rulemaking.

Although EPA received comments about updating the QC acceptance criteria in Method 608.3, EPA did not adopt such changes because EPA lacks data from a multi-laboratory validation study from which to develop such criteria.

F. Change to EPA Method 611

In Section 1.1, EPA corrected the last parameter in the list of parameters table, that read "4-Chlorophenyl phenyl ether," a typographical error. The word "either" should be "ether." The correct parameter name is "4-Chlorophenyl phenyl ether."

G. Changes to EPA Method 624.1

EPA received numerous comments on Method 624.1, ranging from pointing out minor typographical errors to questioning substantive technical aspects of the proposed method. In response, EPA revised the method to address many of those comments. See the response to comments document available in the docket listed in the ADDRESSES section at the beginning of this document for a detailed description of the changes.

Additionally, section 8.1.2.1.2, subsection e, Sample matrices on which MS/MSD tests must be performed for nationwide use of an allowed modification, has been changed to update the web link for the list of industrial categories with existing effluent guidelines to <https://www.epa.gov/cwa-methods/alternate-test-procedure-documents>.

Although EPA received comments about updating the QC acceptance criteria in Method 624.1, EPA did not adopt such changes because EPA lacks data from a multi-laboratory validation study from which to develop such criteria.

H. Changes to EPA Method 625.1

EPA received numerous comments on Method 625.1, ranging from pointing out minor typographical errors to questioning substantive technical aspects of the proposed method. In response, EPA revised the method to address many of those comments. See the response to comments document available in the electronic docket listed in the ADDRESSES section at the beginning of this document for a detailed description of the changes.

Additionally, as was the case with EPA Method 624.1, section 8.1.2.1.2, subsection e, Sample matrices on which MS/MSD tests must be performed for nationwide use of an allowed modification, has been changed to update the web link for the list of industrial categories with existing effluent guidelines to <https://www.epa.gov/cwa-methods/alternate-test-procedure-documents>.

Although EPA received comments about updating the QC acceptance criteria in Method 625.1, EPA did not implement such changes because EPA

lacks data from a multi-laboratory validation study from which to develop such criteria.

I. Changes to Method Detection Limit (MDL) Procedure, Appendix B

No significant revisions were made to the proposed MDL procedure. Some flexibility was added to the procedure, as is discussed in Section II.K above.

J. Changes to WET Errata

Among the corrections that EPA proposed was a change to the language for Fathead minnows, Daphnids, and Green Alga in the document *Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms*, Fourth Edition, U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA/821/R-02/013, October 2002. For Fathead Minnows and Daphnids, EPA proposed to change "Conductivity, alkalinity, and hardness are measured in each new sample (100% effluent or receiving water) and in the control" to read "Conductivity, alkalinity, and hardness are measured at the beginning of the test for all test concentrations in each new sample and in the control before they are dispersed to the test chambers." EPA agrees with commenters that this change would constitute a change to the test rather than a correction or clarification. For that reason, EPA will not add the inserted language "at the beginning of the test for all test concentrations." EPA is retaining its deletion of "(100% effluent or receiving water)" and the insertion of "before they are dispensed to the test chamber" to the end of the sentence. Thus, the sentence will now read "Conductivity, alkalinity, and hardness are measured in each new sample and in the control before they are dispensed to the test chamber." For Green Alga, the proposed change has been eliminated from the errata because only the increased testing was proposed.

IV. Statutory and Executive Order Reviews

A. Executive Order 12866: Regulatory Planning and Review and Executive Order 13563: Improving Regulation and Regulatory Review

This rule is not a "significant regulatory action" under the terms of Executive Order (EO) 12866 (58 FR 51735, October 4, 1993) and is therefore not subject to review under EO 12866 and EO 13563.

B. Paperwork Reduction Act

This action does not impose an information collection burden under the provisions of the Paperwork Reduction

ASTM International for use in compliance monitoring where the Agency has determined that those standards meet the needs of Clean Water Act programs. EPA did not propose to add one Standard Method because that method had not undergone full inter-laboratory validation as recommended in current Agency guidance (see Section IV.C of the proposal for this rule (80 FR 8956, February 19, 2015)). All proposed voluntary consensus standards are approved in this rule.

J. Executive Order 12898: Federal Actions To Address Environmental Justice in Minority Populations and Low-Income Populations

Executive Order (E.O.) 12898 (59 FR 7629 (Feb. 16, 1994)) establishes federal executive policy on environmental justice. Its main provision directs federal agencies, to the greatest extent practicable and permitted by law, to make environmental justice part of their mission by identifying and addressing, as appropriate, disproportionately high and adverse human health or environmental effects of their programs, policies, and activities on minority populations and low-income populations in the United States.

This final rule provides additional compliance methods for use by any facility or laboratory with no disproportionate impact on minority or low-income populations because it merely approves new and revised versions of testing procedures to measure pollutants in water.

K. Congressional Review Act

The Congressional Review Act, 5 U.S.C. 801 *et seq.*, as added by the Small Business Regulatory Enforcement Fairness Act of 1996, generally provides that before a rule may take effect, the agency promulgating the rule must submit a rule report, which includes a copy of the rule, to each House of the Congress and to the Comptroller General of the United States. EPA will submit a report containing this rule and other required information to the U.S. Senate, the U.S. House of Representatives, and the Comptroller General of the United States prior to publication of the rule in the **Federal Register**. This action is not a "major rule" as defined by 5 U.S.C. 804(2). This rule will be effective September 27, 2017.

List of Subjects in 40 CFR Part 136

Environmental protection, Incorporation by reference, Reporting and recordkeeping requirements, Test procedures, Water pollution control.

Dated: August 7, 2017.

E. Scott Pruitt,
Administrator.

For the reasons set out in the preamble, title 40, chapter I of the Code of Federal Regulations is amended as follows:

PART 136—GUIDELINES ESTABLISHING TEST PROCEDURES FOR THE ANALYSIS OF POLLUTANTS

■ 1. The authority citation for part 136 continues to read as follows:

Authority: Secs. 301, 304(h), 307 and 501(a), Pub. L. 95–217, 91 Stat. 1566, *et seq.*

(33 U.S.C. 1251, *et seq.*) (the Federal Water Pollution Control Act Amendments of 1972 as amended by the Clean Water Act of 1977).

■ 2. Section 136.1 is amended by revising paragraph (a) to read as follows:

§ 136.1 Applicability.

(a) The procedures prescribed herein shall, except as noted in §§ 136.4, 136.5, and 136.6, be used to perform the measurements indicated whenever the waste constituent specified is required to be measured for:

(1) An application submitted to the Director and/or reports required to be submitted under NPDES permits or other requests for quantitative or qualitative effluent data under parts 122 through 125 of this chapter; and

(2) Reports required to be submitted by dischargers under the NPDES established by parts 124 and 125 of this chapter; and

(3) Certifications issued by States pursuant to section 401 of the Clean Water Act (CWA), as amended.

* * * * *

■ 3. Section 136.2 is amended by revising paragraphs (d) and (f) to read as follows:

§ 136.2 Definitions.

* * * * *

(d) *Director* means the director as defined in 40 CFR 122.2.

* * * * *

(f) *Detection limit* means the minimum concentration of an analyte (substance) that can be measured and reported with a 99% confidence that the analyte concentration is distinguishable from the method blank results as determined by the procedure set forth at appendix B of this part.

■ 4. In § 136.3:

■ a. Revise paragraph (a) introductory text and tables IA, IB, IC, ID, IF, IG, and IH.

■ b. Revise paragraphs (b) introductory text, (b)(8)(iv), (b)(8)(v), (b)(8)(xiii), (b)(8)(xv), (b)(10)(viii) through (b)(10)(lxvi),

(b)(10)(lxi) through (b)(10)(lxviii), (b)(15)(v), (b)(15)(viii) through (x), (b)(15)(xii), (b)(15)(xiii), (b)(15)(xv) through (xvii), (b)(15)(xxii) through (xxiv), (b)(15)(xxx), (b)(15)(xxxv), (b)(15)(xxxvii), (b)(15)(xxxix), (b)(15)(xlii), (b)(15)(li), (b)(15)(lii), (b)(15)(lv), (b)(15)(lviii), (b)(15)(lix), (b)(15)(lxi), (b)(15)(lxiv), (b)(15)(lxvi), and (b)(15)(lxviii).

■ c. Redesignate paragraphs (b)(19)(vii) and (viii) as paragraphs (b)(19)(ix) and (x), respectively.

■ d. Add new paragraphs (b)(19)(vii) and (viii).

■ e. Revise paragraphs (b)(20)(i) through (iv).

■ f. Remove paragraph (b)(20)(v).

■ g. Revise paragraph (b)(25)(i).

■ h. Add paragraphs (b)(25)(ii) and (iii).

■ i. Redesignate paragraphs (b)(33) and (34) as paragraphs (b)(35) and (36), respectively, and redesignate paragraphs (b)(26) through (32) as paragraphs (b)(27) through (33), respectively.

■ j. Add new paragraphs (b)(26) and (34).

■ k. Revise newly redesignated paragraph (b)(35).

■ l. Revise paragraph (c) and Table II in paragraph (e).

The revisions and additions read as follows:

§ 136.3 Identification of test procedures.

(a) Parameters or pollutants, for which methods are approved, are listed together with test procedure descriptions and references in Tables IA, IB, IC, ID, IE, IF, IG, and IH of this section. The methods listed in Tables IA, IB, IC, ID, IE, IF, IG, and IH are incorporated by reference, see paragraph (b) of this section, with the exception of EPA Methods 200.7, 601–613, 624.1, 625.1, 1613, 1624, and 1625. The full texts of Methods 601–613, 624.1, 625.1, 1613, 1624, and 1625 are printed in appendix A of this part, and the full text of Method 200.7 is printed in appendix C of this part. The full text for determining the method detection limit when using the test procedures is given in appendix B of this part. In the event of a conflict between the reporting requirements of 40 CFR parts 122 and 125 and any reporting requirements associated with the methods listed in these tables, the provisions of 40 CFR parts 122 and 125 are controlling and will determine a permittee's reporting requirements. The full texts of the referenced test procedures are incorporated by reference into Tables IA, IB, IC, ID, IE, IF, IG, and IH. The year after the method number indicates the latest editorial change of the method. The discharge parameter values for which reports are required must be

⁵⁵ Kelada-01, Kelada Automated Test Methods for Total Cyanide, Acid Dissociable Cyanide, and Thiocyanate, EPA 821-B-01-009, Revision 1.2, August 2001. US EPA. Note: A 450-W UV lamp may be used in this method instead of the 550-W lamp specified if it provides performance within the quality control (QC) acceptance criteria of the method in a given instrument. Similarly, modified flow cell configurations and flow conditions may be used in the method, provided that the QC acceptance criteria are met.

⁵⁶ QuikChem Method 10-204-00-1-X, Digestion and Distillation of Total Cyanide in Drinking and Wastewaters using MICRO DIST and Determination of Cyanide by Flow Injection Analysis, Revision 2.2, March 2005. Lachat Instruments.

⁵⁷ When using sulfide removal test procedures described in EPA Method 335.4-1, reconstitute particulate that is filtered with the sample prior to distillation.

⁵⁸ Unless otherwise stated, if the language of this table specifies a sample digestion and/or distillation "followed by" analysis with a method, approved digestion and/or distillation are required prior to analysis.

⁵⁹ Samples analyzed for available cyanide using OI Analytical method OIA-1677-09 or ASTM method D6888-09 that contain particulate matter may be filtered only after the ligand exchange reagents have been added to the samples, because the ligand exchange process converts complexes containing available cyanide to free cyanide, which is not removed by filtration. Analysts are further cautioned to limit the time between the addition of the ligand exchange reagents and sample filtration to no more than 30 minutes to preclude settling of materials in samples.

⁶⁰ Analysts should be aware that pH optima and chromophore absorption maxima might differ when phenol is replaced by a substituted phenol as the color reagent in Berthelot Reaction ("phenol-hypochlorite reaction") colorimetric ammonium determination methods. For example when phenol is used as the color reagent, pH optimum and wavelength of maximum absorbance are about 11.5 and 635 nm, respectively—see, Patton, C.J. and S.R. Crouch. March 1977. Anal. Chem. 49:464-469. These reaction parameters increase to pH > 12.6 and 665 nm when salicylate is used as the color reagent—see, Krom, M.D. April 1980. The Analyst 105:305-316.

⁶¹ If atomic absorption or ICP instrumentation is not available, the aluminum colorimetric method detailed in the 19th Edition of *Standard Methods* may be used. This method has poorer precision and bias than the methods of choice.

⁶² Easy (1-Reagent) Nitrate Method, Revision November 12, 2011. Craig Chinchilla.

⁶³ Hach Method 10360, Luminescence Measurement of Dissolved Oxygen in Water and Wastewater and for Use in the Determination of BOD₅ and cBOD₅, Revision 1.2, October 2011. Hach Company. This method may be used to measure dissolved oxygen when performing the methods approved in Table IB for measurement of biochemical oxygen demand (BOD) and carbonaceous biochemical oxygen demand (CBOD).

⁶⁴ In-Situ Method 1002-8-2009, Dissolved Oxygen (DO) Measurement by Optical Probe. 2009. In-Situ Incorporated.

⁶⁵ Mitchell Method M5331, Determination of Turbidity by Nephelometry. Revision 1.0, July 31, 2008. Leck Mitchell.

⁶⁶ Mitchell Method M5271, Determination of Turbidity by Nephelometry. Revision 1.0, July 31, 2008. Leck Mitchell.

⁶⁷ Orion Method AQ4500, Determination of Turbidity by Nephelometry. Revision 5, March 12, 2009. Thermo Scientific.

⁶⁸ EPA Method 200.5, Determination of Trace Elements in Drinking Water by Axially Viewed Inductively Coupled Plasma-Atomic Emission Spectrometry, EPA/600/R-06/115. Revision 4.2, October 2003. US EPA.

⁶⁹ Method 1627, Kinetic Test Method for the Prediction of Mine Drainage Quality, EPA-821-R-09-002. December 2011. US EPA.

⁷⁰ Techniques and Methods Book 5-B1, Determination of Elements in Natural-Water, Biota, Sediment and Soil Samples Using Collision/Reaction Cell Inductively Coupled Plasma-Mass Spectrometry, Chapter 1, Section B, Methods of the National Water Quality Laboratory, Book 5, Laboratory Analysis, 2006. USGS.

⁷¹ Water-Resources Investigations Report 01-4132, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Organic Plus Inorganic Mercury in Filtered and Unfiltered Natural Water with Cold Vapor-Atomic Fluorescence Spectrometry, 2001. USGS.

⁷² USGS Techniques and Methods 5-B8, Chapter 8, Section B, Methods of the National Water Quality Laboratory Book 5, Laboratory Analysis, 2011. USGS.

⁷³ NECI Method N07-0003, "Nitrate Reductase Nitrate-Nitrogen Analysis," Revision 9.0, March 2014, The Nitrate Elimination Co., Inc.

⁷⁴ Timberline Instruments, LLC Method Ammonia-001, "Determination of Inorganic Ammonia by Continuous Flow Gas Diffusion and Conductivity Cell Analysis," June 2011, Timberline Instruments, LLC.

⁷⁵ Hach Company Method 10206, "Spectrophotometric Measurement of Nitrate in Water and Wastewater," Revision 2.1, January 2013, Hach Company.

⁷⁶ Hach Company Method 10242, "Simplified Spectrophotometric Measurement of Total Kjeldahl Nitrogen in Water and Wastewater," Revision 1.1, January 2013, Hach Company.

⁷⁷ National Council for Air and Stream Improvement (NCASI) Method TNTP-W10900, "Total (Kjeldahl) Nitrogen and Total Phosphorus in Pulp and Paper Biologically Treated Effluent by Alkaline Persulfate Digestion," June 2011, National Council for Air and Stream Improvement, Inc.

⁷⁸ The pH adjusted sample is to be adjusted to 7.6 for NPDES reporting purposes.

TABLE IC—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS

Parameter ¹	Method	EPA ^{2,7}	Standard methods	ASTM	Other
1. Acenaphthene	GC	610			
	GC/MS	625.1, 1625B	6410 B-2000		See footnote, ⁹ p. 27.
	HPLC	610	6440 B-2005	D4657-92 (98)	
2. Acenaphthylene	GC	610			
	GC/MS	625.1, 1625B	6410 B-2000		See footnote, ⁹ p. 27.
	HPLC	610	6440 B-2005	D4657-92 (98)	
3. Acrolein	GC	603			
	GC/MS	624.1, ⁴ 1624B			
4. Acrylonitrile	GC	603			
	GC/MS	624.1, ⁴ 1624B			
5. Anthracene	GC	610			
	GC/MS	625.1, 1625B	6410 B-2000		See footnote, ⁹ p. 27.
	HPLC	610	6440 B-2005	D4657-92 (98)	
6. Benzene	GC	602	6200 C-2011		
	GC/MS	624.1, 1624B	6200 B-2011		
7. Benzidine	Spectro-photometric				See footnote, ³ p. 1.
	GC/MS	625.1 ⁵ , 1625B	6410 B-2000		
	HPLC	605			
8. Benzo(a)anthracene	GC	610			
	GC/MS	625.1, 1625B	6410 B-2000		See footnote, ⁹ p. 27.
	HPLC	610	6440 B-2005	D4657-92 (98)	
9. Benzo(a)pyrene	GC	610			
	GC/MS	625.1, 1625B	6410 B-2000		See footnote, ⁹ p. 27.
	HPLC	610	6440 B-2005	D4657-92 (98)	
10. Benzo(b)fluoranthene	GC	610			
	GC/MS	625.1, 1625B	6410 B-2000		See footnote, ⁹ p. 27.
	HPLC	610	6440 B-2005	D4657-92 (98)	
11. Benzo(g,h,i)perylene	GC	610			
	GC/MS	625.1, 1625B	6410 B-2000		See footnote, ⁹ p. 27.
	HPLC	610	6440 B-2005	D4657-92 (98)	
12. Benzo(k)fluoranthene	GC	610			
	GC/MS	625.1, 1625B	6410 B-2000		See footnote, ⁹ p. 27.
	HPLC	610	6440 B-2005	D4657-92 (98)	
13. Benzyl chloride	GC				See footnote, ³ p. 130.
	GC/MS				See footnote, ⁶ p. S102.
14. Butyl benzyl phthalate	GC	606			
	GC/MS	625.1, 1625B	6410 B-2000		See footnote, ⁹ p. 27.
15. bis(2-Chloroethoxy) methane	GC	611			
	GC/MS	625.1, 1625B	6410 B-2000		See footnote, ⁹ p. 27.
16. bis(2-Chloroethyl) ether	GC	611			
	GC/MS	625.1, 1625B	6410 B-2000		See footnote, ⁹ p. 27.
17. bis(2-Ethylhexyl) phthalate	GC	606			

TABLE IC—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS—Continued

Parameter ¹	Method	EPA ^{2,7}	Standard methods	ASTM	Other
57. Ethylbenzene	GC	602	6200 C-2011		
	GC/MS	624.1, 1624B	6200 B-2011		
58. Fluoranthene	GC	610			
	GC/MS	625.1, 1625B	6410 B-2000		See footnote, ^a p. 27.
	HPLC	610	6440 B-2005	D4657-92 (98)	
59. Fluorene	GC	610			
	GC/MS	625.1, 1625B	6410 B-2000		See footnote, ^a p. 27.
	HPLC	610	6440 B-2005	D4657-92 (98)	
60. 1,2,3,4,6,7,8-Heptachloro-dibenzofuran	GC/MS	1613B			
61. 1,2,3,4,7,8,9-Heptachloro-dibenzofuran	GC/MS	1613B			
62. 1,2,3,4,6,7,8-Heptachloro-dibenzo- <i>p</i> -dioxin	GC/MS	1613B			
63. Hexachlorobenzene	GC	612			
	GC/MS	625.1, 1625B	6410 B-2000		See footnote, ^a p. 27.
64. Hexachlorobutadiene	GC	612			
	GC/MS	625.1, 1625B	6410 B-2000		See footnote, ^a p. 27.
65. Hexachlorocyclopentadiene	GC	612			
	GC/MS	625.1, ⁵ 1625B	6410 B-2000		See footnote, ^a p. 27.
	GC/MS	1613B			
66. 1,2,3,4,7,8-Hexachloro-dibenzofuran	GC/MS	1613B			
67. 1,2,3,6,7,8-Hexachloro-dibenzofuran	GC/MS	1613B			
68. 1,2,3,7,8,9-Hexachloro-dibenzofuran	GC/MS	1613B			
69. 2,3,4,6,7,8-Hexachloro-dibenzofuran	GC/MS	1613B			
70. 1,2,3,4,7,8-Hexachloro-dibenzo- <i>p</i> -dioxin	GC/MS	1613B			
71. 1,2,3,6,7,8-Hexachloro-dibenzo- <i>p</i> -dioxin	GC/MS	1613B			
72. 1,2,3,7,8,9-Hexachloro-dibenzo- <i>p</i> -dioxin	GC/MS	1613B			
73. Hexachloroethane	GC	612			
	GC/MS	625.1, 1625B	6410 B-2000		See footnote, ^a p. 27.
74. Indeno(1,2,3- <i>c,d</i>) pyrene	GC	610			
	GC/MS	625.1, 1625B	6410 B-2000		See footnote, ^a p. 27.
	HPLC	610	6440 B-2005	D4657-92 (98)	
75. Isophorone	GC	609			
	GC/MS	625.1, 1625B	6410 B-2000		See footnote, ^a p. 27.
76. Methylene chloride	GC	601	6200 C-2011		See footnote, ^a p. 130.
	GC/MS	624.1, 1624B	6200 B-2011		
77. 2-Methyl-4,6-dinitrophenol	GC	604	6420 B-2000		
	GC/MS	625.1, 1625B	6410 B-2000		See footnote, ^a p. 27.
78. Naphthalene	GC	610			
	GC/MS	625.1, 1625B	6410 B-2000		See footnote, ^a p. 27.
	HPLC	610	6440 B-2005		
79. Nitrobenzene	GC	609			
	GC/MS	625.1, 1625B	6410 B-2000		See footnote, ^a p. 27.
	HPLC			D4657-92 (98)	
80. 2-Nitrophenol	GC	604	6420 B-2000		
	GC/MS	625.1, 1625B	6410 B-2000		See footnote, ^a p. 27.
81. 4-Nitrophenol	GC	604	6420 B-2000		
	GC/MS	625.1, 1625B	6410 B-2000		See footnote, ^a p. 27.
82. N-Nitrosodimethylamine	GC	607			
	GC/MS	625.1, ⁵ 1625B	6410 B-2000		See footnote, ^a p. 27.
83. N-Nitrosodi- <i>n</i> -propylamine	GC	607			
	GC/MS	625.1, ⁵ 1625B	6410 B-2000		See footnote, ^a p. 27.
84. N-Nitrosodiphenylamine	GC	607			
	GC/MS	625.1, ⁵ 1625B	6410 B-2000		See footnote, ^a p. 27.
85. Octachlorodibenzofuran	GC/MS	1613B ¹⁰			
86. Octachlorodibenzo- <i>p</i> -dioxin	GC/MS	1613B ¹⁰			
87. 2,2'-oxybis(1-chloropropane) ¹² [also known as bis(2-Chloro-1-methylethyl) ether]	GC	611			
88. PCB-1016	GC/MS	625.1, 1625B	6410 B-2000		See footnote, ^a p. 27.
	GC	608.3			See footnote, ^a p. 43; See footnote. ^a
89. PCB-1221	GC/MS	625.1	6410 B-2000		See footnote, ^a p. 43; See footnote. ^a
	GC	608.3			
90. PCB-1232	GC/MS	625.1	6410 B-2000		See footnote, ^a p. 43; See footnote. ^a
	GC	608.3			
91. PCB-1242	GC/MS	625.1	6410 B-2000		See footnote, ^a p. 43; See footnote. ^a
	GC	608.3			
92. PCB-1248	GC/MS	625.1	6410 B-2000		See footnote, ^a p. 43; See footnote. ^a
	GC	608.3			

⁸ Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

⁹ If the sample is not adjusted to pH 2, then the sample must be analyzed within seven days of sampling.

¹⁰ The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.

¹¹ When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity (*i.e.*, use all necessary preservatives and hold for the shortest time listed). When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to $\leq 6^{\circ}\text{C}$, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6–9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (regarding the requirement for thiosulfate reduction), and footnotes 12, 13 (regarding the analysis of benzidine).

¹² If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0 ± 0.2 to prevent rearrangement to benzidine.

¹³ Extracts may be stored up to 30 days at $<0^{\circ}\text{C}$.

¹⁴ For the analysis of diphenylnitrosamine, add 0.008% $\text{Na}_2\text{S}_2\text{O}_3$ and adjust pH to 7–10 with NaOH within 24 hours of sampling.

¹⁵ The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $\text{Na}_2\text{S}_2\text{O}_3$.

¹⁶ Place sufficient ice with the samples in the shipping container to ensure that ice is still present when the samples arrive at the laboratory. However, even if ice is present when the samples arrive, immediately measure the temperature of the samples and confirm that the preservation temperature maximum has not been exceeded. In the isolated cases where it can be documented that this holding temperature cannot be met, the permittee can be given the option of on-site testing or can request a variance. The request for a variance should include supportive data which show that the toxicity of the effluent samples is not reduced because of the increased holding temperature. Aqueous samples must not be frozen. Hand-delivered samples used on the day of collection do not need to be cooled to 0 to 6°C prior to test initiation.

¹⁷ Samples collected for the determination of trace level mercury ($<100\text{ ng/L}$) using EPA Method 1631 must be collected in tightly-capped fluoropolymer or glass bottles and preserved with BrCl or HCl solution within 48 hours of sample collection. The time to preservation may be extended to 28 days if a sample is oxidized in the sample bottle. A sample collected for dissolved trace level mercury should be filtered in the laboratory within 24 hours of the time of collection. However, if circumstances preclude overnight shipment, the sample should be filtered in a designated clean area in the field in accordance with procedures given in Method 1669. If sample integrity will not be maintained by shipment to and filtration in the laboratory, the sample must be filtered in a designated clean area in the field within the time period necessary to maintain sample integrity. A sample that has been collected for determination of total or dissolved trace level mercury must be analyzed within 90 days of sample collection.

¹⁸ Aqueous samples must be preserved at $\leq 6^{\circ}\text{C}$, and should not be frozen unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulatory authority. Also, for purposes of NPDES monitoring, the specification of " $\leq 6^{\circ}\text{C}$ " is used in place of the " 4°C " and " $<4^{\circ}\text{C}$ " sample temperature requirements listed in some methods. It is not necessary to measure the sample temperature to three significant figures ($1/100\text{th}$ of 1°C); rather, three significant figures are specified so that rounding down to 6°C may not be used to meet the $\leq 6^{\circ}\text{C}$ requirement. The preservation temperature does not apply to samples that are analyzed immediately (less than 15 minutes).

¹⁹ An aqueous sample may be collected and shipped without acid preservation. However, acid must be added at least 24 hours before analysis to dissolve any metals that adsorb to the container walls. If the sample must be analyzed within 24 hours of collection, add the acid immediately (see footnote 2). Soil and sediment samples do not need to be preserved with acid. The allowances in this footnote supersede the preservation and holding time requirements in the approved metals methods.

²⁰ To achieve the 28-day holding time, use the ammonium sulfate buffer solution specified in EPA Method 218.6. The allowance in this footnote supersedes preservation and holding time requirements in the approved hexavalent chromium methods, unless this supersession would compromise the measurement, in which case requirements in the method must be followed.

²¹ Holding time is calculated from time of sample collection to elution for samples shipped to the laboratory in bulk and calculated from the time of sample filtration to elution for samples filtered in the field.

²² Sample analysis should begin as soon as possible after receipt; sample incubation must be started no later than 8 hours from time of collection.

²³ For fecal coliform samples for sewage sludge (biosolids) only, the holding time is extended to 24 hours for the following sample types using either EPA Method 1680 (LTB–EC) or 1681 (A–1): Class A composted, Class B aerobically digested, and Class B anaerobically digested.

²⁴ The immediate filtration requirement in orthophosphate measurement is to assess the dissolved or bio-available form of orthophosphorus (*i.e.*, that which passes through a 0.45-micron filter), hence the requirement to filter the sample immediately upon collection (*i.e.*, within 15 minutes of collection).

■ 5. Section 136.4 is amended by revising paragraphs (a) introductory text, (b), and (c) to read as follows:

§ 136.4 Application for and approval of alternate test procedures for nationwide use.

(a) A written application for review of an alternate test procedure (alternate method) for nationwide use may be made by letter via email or by hard copy in triplicate to the National Alternate Test Procedure (ATP) Program Coordinator (National Coordinator), Office of Science and Technology (4303T), Office of Water, U.S. Environmental Protection Agency, 1200 Pennsylvania Ave. NW., Washington, DC 20460. Any application for an ATP under this paragraph (a) shall:

* * * * *

(b) The National Coordinator may request additional information and analyses from the applicant in order to evaluate whether the alternate test

procedure satisfies the applicable requirements of this part.

(c) *Approval for nationwide use.* (1) After a review of the application and any additional analyses requested from the applicant, the National Coordinator will notify the applicant, in writing, of whether the National Coordinator will recommend approval or disapproval of the alternate test procedure for nationwide use in CWA programs. If the application is not recommended for approval, the National Coordinator may specify what additional information might lead to a reconsideration of the application and notify the Regional Alternate Test Procedure Coordinators of the disapproval recommendation. Based on the National Coordinator's recommended disapproval of a proposed alternate test procedure and an assessment of any current approvals for limited uses for the unapproved method, the Regional ATP Coordinator

may decide to withdraw approval of the method for limited use in the Region.

(2) Where the National Coordinator has recommended approval of an applicant's request for nationwide use of an alternate test procedure, the National Coordinator will notify the applicant. The National Coordinator will also notify the Regional ATP Coordinators that they may consider approval of this alternate test procedure for limited use in their Regions based on the information and data provided in the application until the alternate test procedure is approved by publication in a final rule in the **Federal Register**.

(3) EPA will propose to amend this part to include the alternate test procedure in § 136.3. EPA shall make available for review all the factual bases for its proposal, including the method, any performance data submitted by the applicant and any available EPA analysis of those data.

(4) Following public comment, EPA shall publish in the **Federal Register** a

qualitative technique. This method gives analytical conditions for a second GC column that can be used to confirm and quantify measurements. Additionally, Method 625.1 provides gas chromatograph/mass spectrometer (GC/MS) conditions appropriate for the qualitative confirmation of results for the analytes listed in Tables 1 and 2 using the extract produced by this method, and Method 1699 (Reference 18) provides high resolution GC/MS conditions for qualitative confirmation of results using the original sample. When such methods are used to confirm the identifications of the target analytes, the quantitative results should be derived from the procedure with the calibration range and sensitivity that are most appropriate for the intended application.

1.4 The large number of analytes in Tables 1 and 2 makes testing difficult if all analytes are determined simultaneously. Therefore, it is necessary to determine and perform quality control (QC) tests for the "analytes of interest" only. The analytes of interest are those required to be determined by a regulatory/control authority or in a permit, or by a client. If a list of analytes is not specified, the analytes in Table 1 must be determined, at a minimum, and QC testing must be performed for these analytes. The analytes in Table 1 and some of the analytes in Table 2 have been identified as Toxic Pollutants (40 CFR 401.15), expanded to a list of Priority Pollutants (40 CFR part 423, appendix A).

1.5 In this revision to Method 608, Chlordane has been listed as the alpha- and gamma- isomers in Table 1. Reporting may be by the individual isomers, or as the sum of the concentrations of these isomers, as requested or required by a regulatory/control authority or in a permit. Technical Chlordane is listed in Table 2 and may be used in cases where historical reporting has only been the Technical Chlordane. Toxaphene and the PCBs have been moved from Table 1 to Table 2 (Additional Analytes) to distinguish these analytes from the analytes required in quality control tests (Table 1). QC acceptance criteria for Toxaphene and the PCBs have been retained in Table 4 and may continue to be applied if desired, or if these analytes are requested or required by a regulatory/control authority or in a permit. Method 1668C (Reference 17) may be useful for determination of PCBs as individual chlorinated biphenyl congeners, and Method 1699 (Reference 18) may be useful for determination of the pesticides listed in this method. However, at the time of writing of this revision, Methods 1668C and 1699 had not been approved for use at 40 CFR part 136.

1.6 Method detection limits (MDLs; Reference 3) for the analytes in Tables 1 and some of the analytes in Table 2 are listed in those tables. These MDLs were determined in reagent water (Reference 3). Advances in analytical technology, particularly the use of capillary (open-tubular) columns, allowed laboratories to routinely achieve MDLs for the analytes in this method that are 2–10 times lower than those in the version promulgated in 1984. The MDL for an analyte in a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix.

1.6.1 EPA has promulgated this method at 40 CFR part 136 for use in wastewater compliance monitoring under the National Pollutant Discharge Elimination System (NPDES). The data reporting practices described in section 15.6 are focused on such monitoring needs and may not be relevant to other uses of the method.

1.6.2 This method includes "reporting limits" based on EPA's "minimum level" (ML) concept (see the glossary in section 23). Tables 1 and 2 contain MDL values and ML values for many of the analytes.

1.7 The separatory funnel and continuous liquid-liquid sample extraction and concentration steps in this method are essentially the same as those steps in Methods 606, 609, 611, and 612. Thus, a single sample may be extracted to measure the analytes included in the scope of each of these methods. Samples may also be extracted using a disk-based solid-phase extraction (SPE) procedure developed by the 3M Corporation and approved by EPA as an Alternate Test Procedure (ATP) for wastewater analyses in 1995 (Reference 20).

1.8 This method is performance-based. It may be modified to improve performance (e.g., to overcome interferences or improve the accuracy of results) provided all performance requirements are met.

1.8.1 Examples of allowed method modifications are described at 40 CFR 136.6. Other examples of allowed modifications specific to this method are described in section 8.1.2.

1.8.2 Any modification beyond those expressly permitted at 40 CFR 136.6 or in section 8.1.2 of this method shall be considered a major modification subject to application and approval of an alternate test procedure under 40 CFR 136.4 and 136.5.

1.8.3 For regulatory compliance, any modification must be demonstrated to produce results equivalent or superior to results produced by this method when applied to relevant wastewaters (section 8.1.2).

1.9 This method is restricted to use by or under the supervision of analysts experienced in the use of GC/HSD. The laboratory must demonstrate the ability to generate acceptable results with this method using the procedure in section 8.2.

1.10 Terms and units of measure used in this method are given in the glossary at the end of the method.

2. Summary of Method

2.1 A measured volume of sample, the amount required to meet an MDL or reporting limit (nominally 1–L), is extracted with methylene chloride using a separatory funnel, a continuous liquid/liquid extractor, or disk-based solid-phase extraction equipment. The extract is dried and concentrated for cleanup, if required. After cleanup, or if cleanup is not required, the extract is exchanged into an appropriate solvent and concentrated to the volume necessary to meet the required compliance or detection limit, and analyzed by GC/HSD.

2.2 Qualitative identification of an analyte in the extract is performed using the retention times on dissimilar GC columns. Quantitative analysis is performed using the peak areas or peak heights for the analyte on

the dissimilar columns with either the external or internal standard technique.

2.3 Florisil®, alumina, a C18 solid-phase cleanup, and an elemental sulfur cleanup procedure are provided to aid in elimination of interferences that may be encountered. Other cleanup procedures may be used if demonstrated to be effective for the analytes in a wastewater matrix.

3. Contamination and Interferences

3.1 Solvents, reagents, glassware, and other sample processing lab ware may yield artifacts, elevated baselines, or matrix interferences causing misinterpretation of chromatograms. All materials used in the analysis must be demonstrated free from contamination and interferences by running blanks initially and with each extraction batch (samples started through the extraction process in a given 24-hour period, to a maximum of 20 samples—see Glossary for detailed definition), as described in section 8.5. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required. Where possible, labware is cleaned by extraction or solvent rinse, or baking in a kiln or oven.

3.2 Glassware must be scrupulously cleaned (Reference 4). Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. Solvent rinsing should be followed by detergent washing with hot water, and rinses with tap water and reagent water. The glassware should then be drained dry, and heated at 400 °C for 15–30 minutes. Some thermally stable materials, such as PCBs, may require higher temperatures and longer baking times for removal. Solvent rinses with pesticide quality acetone, hexane, or other solvents may be substituted for heating. Do not heat volumetric labware above 90 °C. After drying and cooling, store inverted or capped with solvent-rinsed or baked aluminum foil in a clean environment to prevent accumulation of dust or other contaminants.

3.3 Interferences by phthalate esters can pose a major problem in pesticide analysis when using the electron capture detector. The phthalate esters generally appear in the chromatogram as large late eluting peaks, especially in the 15 and 50% fractions from Florisil®. Common flexible plastics contain varying amounts of phthalates that may be extracted or leached from such materials during laboratory operations. Cross contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Interferences from phthalates can best be minimized by avoiding use of non-fluoropolymer plastics in the laboratory. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination (References 5 and 6). Interferences from phthalate esters can be avoided by using a microcoulometric or electrolytic conductivity detector.

3.4 Matrix interferences may be caused by contaminants co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the industrial complex or municipality being sampled. Interferences extracted from

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